

June 29, 1936

PREPARATION OF A SPECIAL PINE TAR OIL, Sp. Gr. 1.068
(Pine-Trel) (Dr. McGovern)

The American Turpentine and Tar Company, New Orleans, La. prepares a special grade of pine tar oil, sp. gr. 1.068, for use in treatment of wounds on animals. This material is marketed under the trade name of "Pine-Trel". It differs from the usual run of pine tar oil made in the plant of the American Turpentine and Tar Company in that it is filtered which removes solids and facilitates dehydration; also the excess acid in the special pine tar oil is neutralized with sodium hydroxide.

Mr. C.E. Meriwether, Jr., who has charge of production in the American Turpentine and Tar Company Plant at New Orleans, La., explained their methods of production in detail and showed me the equipment that they use. He pointed out that in their plant all the metal which comes in contact with the pine tar oil during the entire process of preparation is copper or brass. He considers this point to be very important in the manufacture of pine tar oil. Mr. Meriwether gave me all the information he had available and made many helpful suggestions about the constituents and properties of their pine tar oil and pine tar oil in general. He gave me almost a full day of his time and expressed himself as willing to cooperate with the Bureau of Entomology and Plant Quarantine in any way he could. Mr. Meriwether did not request that the information he furnished be kept confidential but probably it should not be given out to persons outside the Bureau of Entomology and Plant Quarantine without his permission.

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Outline of Process of Making the Special Pine Tar Oil 1.065 (Pine-trel)

1. Material. Heart wood, "fat pine" or "lighter wood" from long leaf yellow pine grown in Mississippi or Louisiana was used. Five or ten years after the logs suitable for lumber have been removed, the sap wood has rotted off the branches, knots, and tree tops that were left on the ground. This heart wood averaged about 6 to 8 inches in diameter and was cut in lengths convenient for loading into box cars. Considerable rotten sap wood and dirt adhered to the outside of these pieces of heart wood. No stumps were used.
2. Retort for Heating Wood. Two and one half cords of heart wood of long leaf yellow pine were placed in each retort. The retort was closed and heated gradually to a maximum of 1250 to 1300° F. with a natural gas fire applied to the outside of the retort. No fire was permitted to enter the retort. The vapor that formed in each retort was condensed by passing through a nozzle and into a water-cooled condenser that consisted of ninety 1-1/4" pipes each about 5 feet long. The water for cooling the condenser was pumped from a canal that adjoined the plant. Enough water was run through the condenser to cool it sufficiently to liquify all condensable material so that no vapor passed through the pipes into the storage tanks. The non-condensable gases were drawn off from the condenser and burned. A water trap in this line with 4 to 6 inches of water in it regulated the pressure in the retort so that a pressure equal to 4 to 6 inches of water was maintained in the retort. No non-volatile material went into the pine tar oil.

Outline of Process of Making the Special Pine Tar Oil (Pine-Tar)

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2. Retort for Heating Wood. Two and one half cords of heart wood of long leaf yellow pine were placed in each retort. The retort was closed and heated gradually to a maximum of 180° F. with a natural gas fire applied to the outside of the retort. No fire was permitted to enter the retort. The vapor that formed in each retort was condensed by passing through a nozzle and into a water-cooled condenser that consisted of ninety 1-1/4" pipes each about 5 feet long. The water for cooling the condenser was pumped from a canal that adjoined the plant. Enough water was run through the condenser to cool it sufficiently to lightly oil condensable material so that no vapor passed through the pipes into the storage tanks. The non-condensable gases were drawn off from the condenser and burned. A water trap in this line with 4 to 6 inches of water in it regulated the pressure in the retort so that a pressure of 1 to 2 inches of water was maintained in the retort. No non-volatile material went into the pine tar oil.

The non-volatile residue remained in the retort as charcoal.

3. Separation of Pine Tar Oil from Pyroligneous Acid. The liquids

from the retort condenser were pumped into large tanks. This crude material was made into two cuts. In the first cut the pine tar oil was lighter than the pyroligneous acid and in the second cut the pine tar oil was heavier than the pyroligneous acid.

The specific gravity of pyroligneous acid was from 1.020 to 1.035.

These cuts were made to facilitate the separation of the pyroligneous acid from the pine tar oil. These cuts were allowed to stand and the pine tar oil to separate from the pyroligneous acid which was then drawn off. The pine tar oil sp. gr. 1.065 that was especially prepared for wound treatment was made from the first cut of the crude material.

4. Filtering. Pine tar oils are not filtered unless requested

by the buyer. The cordage trade requires that the pine tar oil they use be filtered. After the gravity separation of pyroligneous acid has been completed the crude pine tar oil was filtered.

Powdered charcoal was added to the pine tar oil and the mixture was pumped through a filter of twill cloth with a layer of manila paper over it. The pine tar oil was pumped through the filter at 45 lbs. pressure and 50° C. This filtering process removed the solids, some sticky materials of various kinds, and facilitated the removal of any pyroligneous acid that was still in the pine tar oil by increasing the size of the drops of acid in the oil.

The non-volatile residue remained in the retort as charcoal.

3. Separation of Pine Tar Oil from Pyroigneous Acid. The mixture

from the retort condenser were pumped into large tanks. This crude material was made into two cuts. In the first cut the pine tar oil was lighter than the pyroigneous acid and in the second cut the pine tar oil was heavier than the pyroigneous acid.

The specific gravity of pyroigneous acid was from 1.030 to 1.035.

These cuts were made to facilitate the separation of the pyroigneous acid from the pine tar oil. These cuts were allowed to stand and the pine tar oil to separate from the pyroigneous acid which was then drawn off. The pine tar oil sp. gr. 1.035 that was especially prepared for kerosene treatment was made from the

first cut of the crude material.

4. Filtering. The tar oils are not filtered unless requested

by the buyer. The condenser residue that the pine tar oil they use be filtered. After the gravity separation of pyroigneous acid has been completed the crude pine tar oil was filtered.

Forced charcoal was added to the pine tar oil and the mixture

was pumped through a filter of twill cloth with a layer of

hemp paper over it. The pine tar oil was pumped through the

filter at 45 lbs. pressure and 80°. This filtering process

removed the solids, some sticky materials of various kinds, and

facilitated the removal of any pyroigneous acid that was still

in the pine tar oil by increasing the size of the drops of acid

in the oil.

The pine tar oil was then pumped into large tanks.

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5. Neutralization of Acids Remaining in the Pine Tar Oil. After filtration sodium hydroxide was added to the pine tar oil until wash water from the oil was neutral to litmus. "Pine-Trel" was the only pine tar oil made by the American Turpentine and Tar Company that received this neutralizing treatment.
6. Final Distillation. The neutralized pine tar oil was then put in a retort heated with a steam coil. The steam was at 125 lbs. pressure at the boiler. A rather slow distillation (about 1/2 to 3/4 gallons per minute) was run until the amount of distillate coming over began to fall off. Live steam was then released in the retort through a jet and the distillation continued until the pine tar oil that remained in the retort reached a specific gravity of 1.065. This pine tar oil was the finished product and was put in drums for shipment.

3. Neutralization of Acids Remaining in the Pine Tar Oil. After filtration sodium hydroxide was added to the pine tar oil until wash water from the oil was neutral to litmus. "Pine-Tar" was the only pine tar oil made by the American Turpentine and Tar Company that retained this neutralizing treatment.
4. Final Distillation. The neutralized pine tar oil was then put in a retort heated with a steam coil. The steam was at 125 lbs. pressure at the bellows. A rather slow distillation (about 1/2 to 3/4 gallons per minute) was run until the amount of distillate coming over began to fall off. Live steam was then released into the retort through a jet and the distillation continued until the pine tar oil that remained in the retort reached a specific gravity of 1.065. This pine tar oil was the finished product and was put in drums for shipment.
5. Distillation of the Pine Tar Oil. The pine tar oil was then put in a retort heated with a steam coil. The steam was at 125 lbs. pressure at the bellows. A rather slow distillation (about 1/2 to 3/4 gallons per minute) was run until the amount of distillate coming over began to fall off. Live steam was then released into the retort through a jet and the distillation continued until the pine tar oil that remained in the retort reached a specific gravity of 1.065. This pine tar oil was the finished product and was put in drums for shipment.

General Notes on Pine Products Made by American Turpentine and Tar Co.

The products, except the non-condensable gases which were burned, that were vaporized from the heart wood of pine in the retort were condensed and redistilled. However, before this second distillation most of the pyroligneous acid was separated from the crude pine tar oil by gravity.

From this second distillation of the crude pine tar oil the following products were obtained:

A. Distillates listed in the order they distilled over:

1. "Lacksol" - a wood naphtha.
2. "Pinap" (not always separated out).
3. Turpentine.
4. Commercial Dipentene.
5. Pine Oil - all the pine oil is put in one grade.

B. Residue that remained after this second distillation:

1. Pine tar oil.
If this second distillation were continued an oil, called "Double X" Oil by Mr. Meriwether, was distilled over and the material that remained in the retort at the end was pitch which was a solid at outdoor temperature.

Mr. Meriwether made a rough estimate of the main constituents removed from pine tar oil of different specific gravities.

1. Pine tar oil, sp. gr. 1.0.
This contained everything distilled from the heart wood of pine except the non-condensable gases and the pyroligneous acid.
2. Pine tar oil, sp. gr. 1.035.
All the "Lacksol" and 90% of the turpentine have been removed.
3. Pine tar oil, sp. gr. 1.065.
All the "Lacksol", all the turpentine and 15 to 20% of the pine oil have been removed.

The products, except the non-condensable gases which were burned, that were vaporized from the heart wood of pine in the retort were condensed and redistilled. However, before this second distillation most of the pyroligneous acid was separated from the crude pine tar oil by gravity.

From this second distillation of the crude pine tar oil the following products were obtained:

A. Distillates listed in the order they distilled over:

1. "Lackacol" - a wood naphtha.
2. "Pine" (not always separated out).
3. Turpentine.
4. Commercial kerosene.
5. Pine Oil - all the pine oil is not in one grade.

B. Residue that remained after this second distillation:

1. Pine tar oil.
If this second distillation were continued an oil called "Residue K" Oil by Mr. Marivether, was distilled over and the material that remained in the retort at the end was pitch which was a solid at outdoor temperature.

Mr. Marivether made a rough estimate of the main constituents removed from pine tar oil of different specific gravities.

1. Pine tar oil, sp. gr. 1.0.
This contained everything distilled from the heart wood of pine except the non-condensable gases and the pyroligneous acid.
2. Pine tar oil, sp. gr. 1.025.
All the "Lackacol" and 30% of the turpentine have been removed.
3. Pine tar oil, sp. gr. 1.055.
All the "Lackacol", all the turpentine and 15 to 20% of the pine oil have been removed.

4. Pine tar oil, sp. gr. 1.075.
All the "Lacksol", all the turpentine and 90% of the pine oil have been removed.
5. Pine tar oil, sp. gr. 1.085.
All the "Lacksol", all the turpentine, all the pine oil and a small part of the "Double X" oil have been removed.

Pine tar oils with specific gravities of 1.065, or less, were made from the first cut to come over from the wood in the retort. This crude pine tar oil was lighter than pyroligneous acid.

Pine tar oil with specific gravities greater than 1.065 were made from the second cut to come from the wood. This crude pine tar oil was heavier than pyroligneous acid.

The following specifications were for the American Turpentine and Tar Company regular pine tar oil, specific gravity 1.065.

Similar specifications for "Pine-Trol" were not available.

Viscosity - Saybolt, Furol - 20 to 25 seconds at 50° C.
Viscosity - Stormer - 90 to 180 seconds at 30° C.
Acid number 50.6 (approximately).
Rosin content 19% "
Flash point 220° F.
Fire Point 238° F.
Iodine number -Wijs Method - 130.7 (approximately).
Saponification number 68 (approximate).
Specific gravity 1.065 $\pm$ or - 0.005.

The regular grades of pine tar are named as follows:

1. Thin pine tar, sp. gr. 1.065 $\pm$ or - 0.005
2. Medium pine tar, sp. gr. 1.075 $\pm$ or - 0.005.
3. Heavy pine tar, sp. gr. 1.085 $\pm$ or - 0.005.

4. Pine tar oil, sp. gr. 1.075.
All the "blackoil", all the turpentine and 80% of the pine
oil have been removed.

5. Pine tar oil, sp. gr. 1.065.
All the "blackoil", all the turpentine, all the pine oil
and a small part of the "double X" oil have been removed.

Pine tar oils with specific gravities of 1.055, or less, were

made from the first cut to come over from the wood in the forest.

This crude pine tar oil was lighter than pyroligneous acid.

Pine tar oil with specific gravities greater than 1.055 were

made from the second cut to come from the wood. This crude pine tar

oil was heavier than pyroligneous acid.

The following specifications were for the American Turpentine

and Tar Company regular pine tar oil, specific gravity 1.065.

Similar specifications for "Pine-Tarol" were not available.

Viscosity - Saybolt, Turcol - 30 to 35 seconds at 50° C.

Viscosity - Stormer - 30 to 180 seconds at 30° C.

Acid number 30.5 (approximately).

Resin content 13%

Flash point 230° F.

Pire point 238° F.

Iodine number - With method - 130.7 (approximately).

Specification number 58 (approximate).

Specific gravity 1.065 ± or - 0.005.

The regular grades of pine tar are named as follows:

1. Thin pine tar, sp. gr. 1.065 ± or - 0.005

2. Medium pine tar, sp. gr. 1.075 ± or - 0.005.

3. Heavy pine tar, sp. gr. 1.085 ± or - 0.005.

4. Pine tar oil, sp. gr. 1.075.
All the "blackoil" and 80% of the turpentine have been removed.

5. Pine tar oil, sp. gr. 1.065.
All the "blackoil", all the turpentine and 10% of the
pine oil have been removed.

TYPICAL DISTILLATION RANGE OF PINE TAR AND PINE TAR OIL MADE BY
AMERICAN TURPENTINE AND TAR COMPANY.

	Commercial Tar Oil	Thin Tar	Medium Tar	Heavy Tar
5% -----	160° C -----	211° C -----	225° C -----	242° C -----
10% -----	193.5 -----	221 -----	243 -----	260 -----
15% -----	215 -----	236 -----	260 -----	271 -----
20% -----	224 -----	246 -----	272 -----	292 -----
25% -----	232 -----	262 -----	288 -----	312 -----
30% -----	237 -----	292 -----	301 -----	334 -----
35% -----	249 -----	315 -----	316 -----	342 -----
40% -----	264 -----	328 -----	332 -----	346 -----
45% -----	281 -----	356 -----	340 -----	361 -----
50% -----	309.5 -----	340 -----	354 -----	370 -----
55% -----	327 -----	344 -----	366 -----	
60% -----	336 -----	360 -----	370 -----	
65% -----	342 -----	366 -----		
70% -----	353 -----	370 -----		
75% -----	358 -----			
80% -----	360 -----			

The above figures are approximate -
slight tolerance (plus or minus)
permitted.

AMERICAN TURPENTINE AND TAR COMPANY.
TYPICAL DISTILLATION RANGE OF THE TARS AND TAR OIL MADE BY

Heavy Tar	Medium Tar	Thin Tar	Commercial Tar Oil
245°	255°	211°	166°
260	263	221	183.5
271	280	236	215
283	278	246	224
273	288	252	233
284	301	257	237
292	316	315	249
306	332	326	264
321	340	336	281
330	354	340	309.5
	366	344	327
	370	350	336
		356	342
		370	353
			358
			360

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In the discussion of the use of pine tar on animals it was brought out that the crude pine tar often caused the skin of animals to crack and bleed. Mr. Meriwether suggested that this might be due to the oxidation of the pine tar forming a hard surface which would "check" or crack. The cracks that formed in the pine tar that was stuck tightly to the hair and skin of the animal would open with sufficient force to break the skin and thus cause bleeding. He suggested the addition of some material to reduce this cracking or "checking". He stated that dipentene was used to prevent "checking" in varnish.

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The above report by Mr. Meriwether is being submitted for your information.